Analysis for Failure, Damage, and Fracture

1 Introduction

Failure analysis is a long-established practice with its roots in all of the engineering sciences. In fact, modern industry could not exist in its present form without failure analysis being used to advance technology. Metallurgy in particular is a mature engineering field extensively researched and covering a broad spectrum of subjects. Failure analysis examinations use this vast knowledge to try to determine why a metal component failed to perform adequately in use.

2 Scope

This document applies to caseworking personnel using the associated instrument(s)/equipment in support of metallurgy examinations. The field of metallurgical failure analysis encompasses all of metallurgy and materials science and engineering, from raw material production to end product use. There is an extremely wide variety of types of parts, alloys, post-manufacture treatments, service conditions, types of loading, applications, environments, and combinations of all of these. In addition, there are nearly unlimited questions that can be asked and determinations that can be requested with regard to the failure and/or damage exhibited.

The metallurgical failure analysis practitioner recognizes that each examination and testing procedure is situational and requires sound engineering judgment and discretion. Not all examination steps in this procedure are required in every case but several techniques are listed for examiner consideration. This procedure outlines analyses which may be performed in examination(s) for failure/damage/fractures. The standard operating procedures (SOPs) for each of the instruments used for a given set of examinations should also be consulted.

3 Principle

Failure analysis is fundamentally an engineering-based investigation of an event or a series of events. By application of engineering principles, an item can be examined to determine if fundamental design problems exist or if a defect may have been artificially introduced. For example, a sharp change in cross-section can result in a stress concentration and crack development under conditions not otherwise expected for the assumed operating stresses. Fracture mechanics permits analysis of such a stress concentration to predict its potential effects on the component's operating life.

Fractography can be applied to determine the mechanism responsible for crack growth and to determine the crack initiation site. Some fracture mechanism examples include simple tensile overload, fatigue, stress corrosion cracking, and creep failure. Each fracture surface also provides information on the nature of the applied stress (i.e., torsional, shear, tensile, mixed mode). The

metallographic and/or compositional analysis of an object may reveal deficiencies in material or manufacturing process as a cause of failure. Numerous other methodologies are also available for exploring a particular aspect of a failure and determining its cause.

4 Specimens

Nearly any metal object and many nonmetallic objects can be examined using the procedures outlined in this protocol.

5 Equipment/Materials/Reagents

A list of items commonly used in these examinations follows. Not every item is used for all failure and damage investigations. The instrumentation and equipment to be employed will depend on the nature of the items to be examined. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) for additional supplies (see 15 References).

a. Photography equipment for macro- and micro-documentation

b. Observation enhancing tools, such as:
   i. borescope, magnifying glass, jewelers' loupe
   ii. visible light microscopes (stereomicroscope, digital microscope)
   iii. scanning electron microscope (SEM)

c. Radiography system*

d. Measurement tools, such as:
   i. micrometers, calipers, measuring tape
   ii. optical measuring microscope (e.g., SmartScope FOV*)
   iii. balances
   iv. magnet

e. Miscellaneous hand tools

f. Certified reference materials (CRMs), reference materials, and standardization materials as needed

g. Digital multimeter

h. Specimen cleaning and protection equipment and materials:
   i. compressed air
   ii. lint free wipes

iii. cleaning brushes
iv. cellulose acetate replication tape
v. EvapoRust™ rust remover
vi. Solvents: water, alcohol, etc.
vii. ultrasonic cleaner
viii. desiccant
ix. vacuum chamber

i. Compositional analysis equipment:
   i. Energy dispersive x-ray fluorescence spectrometer (EDXRF)*
   ii. Spark discharge-in-argon optical emission spectrometer (SDAR-OES)*
   iii. Scanning electron microscope with energy dispersive x-ray spectrometer (SEM/EDS)

j. Metallographic specimen preparation and examination equipment*

k. Non-destructive testing equipment, such as:
   i. magnetic particle inspection equipment
   ii. liquid dye penetrant (LDP) and developer
   iii. ultrasonic inspection equipment

l. Mechanical testing equipment, such as:
   i. Hardness* and microhardness* testers
   ii. Tensile*, torsion, fatigue, impact and wear testers

6 Standards and Controls

The standards and control materials to be employed in this procedure will depend on the specific analytic methods employed and the nature of the items under analysis. Any instrument used in this procedure will use the standards required under its specific SOP. Exemplars for evidentiary items will be obtained as needed.

7 Sampling

Visual examinations are performed on every item examined under this protocol. Further testing is based on the suitability of individual items, or portions of items, for relevant examination techniques. Case notes will describe which examinations were performed on which items. If initial examinations reveal that an analyzed characteristic may vary on a single item, the means of selecting a location to test the characteristic will be noted in the case file.

If an item contains multiple visually indistinguishable objects (e.g., IED container fragments) that are suitable for one analysis technique, a subset may be selected for testing by (1) non-statistical

or (2) statistical means. Any sampling plan and corresponding procedure used will be recorded in case notes.

(1) For non-statistical specimen selection, the report will attribute the measured characteristic only to the specimen(s) tested. This can be facilitated by sub-dividing the evidence and reporting the specific analysis results for the sub-divided portion only.

(2) If a sampling plan will be used to make an inference about the entire set of visually similar items, then the plan will be based on a statistically valid approach. A hypergeometric distribution can be used to describe the probability of encountering deviations within a set of items when not every item is tested. (See Appendix A.) Appendix A assumes that all results are consistent. If inconsistent results are encountered, metallurgy conclusions regarding that characteristic will be limited to the specimens tested.

8 Procedure

The following analysis sequence was derived from guidelines established by ASM International and augmented for forensic metallurgical applications. Each of the listed analyses are not required in every situation. Further, this protocol will not be taken as a substitute for sound engineering judgment. Data gathered during examinations will be included in the case notes.

a. Perform a preliminary visual and low magnification microscopic evaluation of the nature of comparison or item, fabrication, coating, service use/abuse, type(s) of failure, possible contamination, and any other characteristics deemed valuable. This preliminary exam serves to formulate a general concept for the approach to examination, sampling, and testing. Care should be exercised to ensure that mating fracture surfaces are not brought into contact with each other to “see if they fit” to avoid possible destruction of valuable surface information.

b. Photograph the submitted or in situ items in the “as-received condition” (ARC) prior to any extraction or retrieval for laboratory examinations. These photographs will record component positions, in situ conditions, fracture and failure orientation relative to its environment and to other components, service conditions, service abuse, and any other characteristic, condition or information to be considered during the failure/damage analysis. Whenever practicable, include a scale in the photograph or apply a verified micron marker to the photograph. Additional photographs can be taken during various stages of the examinations to record more detailed characteristics of morphology, microstructure, contaminants, or other features relevant to the analysis. If the evidence is altered for examination, note the modification in the case notes.

c. Evaluate the physical properties of the items by measuring dimensions, mass and magnetic response.

d. Sample selection should be conducted causing as little damage as possible. Record with notes and photographs any information derived from the preliminary examinations which may potentially be needed to reach a conclusion but could be considered damaged or eliminated due to sample or specimen removal. Provide adequate protection of all fracture surfaces and damaged regions to prevent contact with each other, with other portions of the same component, or with other objects or items during transport and/or examination. If appropriate and feasible, package with desiccant to reduce degradation by corrosion.

e. If available and appropriate, acquire and analyze documentary information to assist in the reconstruction of the sequence of events leading to the damage and/or failure. While such documentation can help to narrow the focus of an analysis and provide useful guidance, it is not a substitute for the physical evidence generally required to definitively establish a failure mode. Ordinarily, such information is collected by the submitting agency and may include fabrication, manufacturing and processing information; service history; interviews of eyewitness individuals; interviews with individuals whose duties, behavior or failure to act may have induced, or may have affected, the material behavior in question; as received item photos; site/in situ photographs; repair history; environmental details (e.g., temperatures, loading conditions, load magnitude(s), environment chemistry); and similar component history.

f. Prior to any specimen cleaning, perform visual and low power magnification examinations of fracture surfaces, secondary cracks, relevant surface phenomena, gross deformation, thermal damage, and any other metallurgical or environmental characteristic deemed appropriate.

g. If appropriate and feasible, specimens should be taken from the items for chemical analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested. It may be necessary to do some or all specimen retrieval prior to, or between stages of, cleaning. Analyses such as EDXRF and SEM/EDS can often be helpful in identifying the chemical compounds present on a failed component.

h. If appropriate, clean specimen(s) using methods that progress from least to most aggressive (see Table 1 for examples) until contaminant is removed. Preserve any replica(s) or contaminant/debris for appropriate analysis (see 8.g).

Table 1: Specimen Cleaning Procedures	
Increasingly Aggressive ↓	Dry air blast
	Soft artist's brush
	Stiff non-metallic brush
	Aqueous rinse
	Organic solvent (e.g., isopropanol, hexane)
	Chelating Agent (EvapoRust™)
	Ultrasonic cleaning
	Plastic replication

a. After cleaning, perform additional visual and low power magnification examinations of fracture surfaces and other relevant metallurgical characteristics.

b. As needed, stereomicroscopic and SEM examinations may be performed to further characterize fracture surface features and any exogenous material present.

c. Nondestructive testing (i.e., magnetic particle, LDP, ultrasonic, x-ray, and other electromagnetic evaluations) may be performed following the appropriate instrument SOP and protocol. Non-destructive testing can find unopened cracks. Such cracks can sometimes be used to make inferences about the failure mode when the fracture surfaces are too damaged to allow interpretation. They can also be used to determine how widespread a failure mechanism is in a given system. For example, a fracture in a gas pipeline may be accompanied by other, unopened cracks. If unrepaired, these can potentially lead to additional failures following repair of the known defect.

d. As appropriate, destructive testing (i.e., hardness, tensile, impact testing) may be conducted to characterize mechanical and material properties.

e. If needed, metallographic examinations may be performed either before or after mechanical testing for the evaluation of inclusions; microstructural segregation or inhomogeneities; decarburization; carbon pick-up; improper heat treatment; corrosion; grain size; type, distribution and morphology of microstructural constituent(s); or any other characteristics present of metallurgical interest.

f. Supplementary examinations of metallographic specimens by SEM/EDS can be used to further characterize phase distribution and identify elements present in different microstructural regions of interest.

g. An evaluation of the data and facts accumulated from the above analyses performed should allow for the determination of the fracture mode or cause(s) of the damage exhibited.

h. If appropriate, a mathematical analysis of mechanical factors leading to fracture may be used to:
   i. predict flaw size which caused catastrophic fracture at a load below that expected to cause failure
   ii. evaluate manufacturing flaws
   iii. establish a quantitative framework for evaluating structural reliability
   iv. assist in the design and prediction of service life

i. Consideration should be given to testing similar specimens (exemplars) under simulated conditions when the history and service conditions of the questioned specimen are known.

j. Report findings after evaluation of all gathered data.

k. Although it is not typical for criminal cases, it may be prudent to suggest corrective measures to prevent future failures.

9 Instrumental Conditions

For instruments that require verification, standardization or energy adjustment, a copy of the appropriate record(s) will be included in the case notes.

9.1 Analytical Instruments

For the instruments noted (*) in the Equipment/Materials/Reagents section, follow the appropriate CU Metallurgy SOP (see 15 References).

9.2 Supporting Equipment

The following additional instrumental conditions also will be applied:

a. Macro-and micro-photographs will contain a reference scale whenever feasible, however these are included for general reference and measurements will not be made from the images. Micron markers that are automatically generated by camera or microscope

software are to be considered approximate and also will not be used to measure features within the image unless the marker is verified against a calibrated scale.

b. When possible, cutting and grinding operations will be lubricated to prevent overheating that can change the metallurgical characteristics of the specimen. If lubrication is not possible, the metallurgical changes imparted by the process must be considered during analysis.

c. The following instruments will be verified according to the appropriate CU Instrument Operation & Systems Support (IOSS) SOP (see 15 References) prior to their first use to acquire case data on any given day:
    i. traceable micrometers/calipers
    ii. traceable balances

9.3 SEM/EDS

Compositional analysis by SEM/EDS will be conducted as follows:

a. Prior to their first use to acquire case data on any given day, run the instrument performance verification routine according to the appropriate IOSS SOP (see 15 References). File one copy with the instrument performance records.

b. Prepare and insert the specimen(s) ensuring electrical continuity with the specimen stage.

c. Adjust the instrument conditions to image the region of interest for analysis. Backscattered electron imaging can be helpful to locate features that differ in mean atomic number from their surroundings.

d. Acquisition duration will depend on the conditions chosen and the specimen area exposed to the incident beam. The acquisition time can be extended to optimize spectrum clarity or shortened to enhance collection efficiency based on the case requirements.

e. Label the elemental peaks on the acquired spectrum, considering peak shapes and energy positions, the relative heights of adjacent peaks and system generated peaks. Many SEM/EDS systems have software that can accurately identify the escape and sum peaks in a spectrum. The peak identification system resident in the instrument software can be augmented by analyzing CRMs of similar composition to the specimen of interest.

f. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.

10 Decision Criteria

The conclusions derived from this procedure are based on careful interpretation of all factual information gathered from completed testing and investigation. If a unique scenario does not explain the failure, all possibilities deemed relevant by the examiner will be reported in the conclusion. Conclusions will be expressed in reports and testimony according to current FBI Laboratory requirements (see the References section).

Analysis for failure, damage, and fracture contributes to evaluating whether two or more items or materials were once part of the same object. 'Fracture fit' is an examiner's conclusion that two or more metallurgy items or materials were once part of the same object. This conclusion is an examiner's decision that two or more metallurgy items or materials show sufficient correspondence between their observed characteristics to indicate that they once comprised a single object and insufficient disagreement between their observed characteristics to conclude that they originated from different objects. This conclusion can only be reached when portions of two or more metallurgy items or materials physically fit together.

11 Calculations

A wide range of possible calculations can be encountered in a failure analysis. These are case-specific and may include determination of the applied stresses and strains, fracture mechanics calculations, and corrosion-related calculations. The references listed in the References section contain useful information sources for these calculations.

12 Measurement Uncertainty

When gathered, quantitative data are generally used for comparative purposes as described in *Examinations for Association or Origin*. Expanded measurement uncertainty should not be used for these inter-comparisons because it increases the probability two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion). Instead, the variances associated with the samples and with data acquisition are accommodated by the statistical comparison.

If it should be necessary to estimate the measurement uncertainty of an instrumental result, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*. Instrumental measurement uncertainty is addressed in the individual instrument SOPs and will be calculated and reported when appropriate. For mechanical testing and compositional analysis instruments, each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated. For calibrated, traceable dimensional measuring equipment, the repeatability component will be updated annually.

13 Limitations

The limitations of a particular failure analysis are determined by the type, amount and condition of object(s) being analyzed; the available background information; the specific examinations required; and subsequent determinations made, and therefore cannot be predicted within this protocol but will be reported in the case notes.

14 Safety

a. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.

b. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review instrument SOPs and pertinent material Safety Data Sheets (SDS) prior to conducting examinations. If additional guidance is required, contact the Laboratory Health and Safety Group.

c. Mechanical hazards in the form of mechanical testing equipment and other machinery may require special precautions**.** Refer to the equipment manufacturer's guidelines regarding personal safety protocol and/or consult the Laboratory Health and Safety Group.

15 References

ASM International Failure Analysis Committee, *Handbook of Case Histories in Failure Analysis,* ASM International 1992

Wulpi, D. J., *Understanding How Components Fail,* 2nd edition, ASM International 1999

ASM International, Materials Engineering Institute, *General Procedures for Failure Analysis, Course 13, Lesson 1*, ASM International 1990 (videotape)

Jones, D. A., *Principles and Prevention of Corrosion*, 2nd edition, Prentice-Hall Inc. 1996

Pletcher, D. and Walsh, F. C., *Industrial Electrochemistry,* 2nd edition*,* Chapman and Hall 1990

Cartz, L., *Nondestructive Testing*, ASM International 1995

Anderson, R. C., *Inspection of Metals, Volume I: Visual Examination*, American Society for Metals 1983

Shames, I. H. and Pitarresi, J. M., *Introduction to Solid Mechanics,* 3rd edition*,* Prentice-Hall Inc. 2000

Hertzberg, R. W., Vinci, R. P. and Hertzberg, J. L., *Deformation and Fracture Mechanics of Engineering Materials*, 5th edition, John Wiley & Sons, Inc. 2012

Hibbeler, R. C., *Mechanics of Materials, 4th edition,* Prentice-Hall Inc. 2000

Anderson, T. L., *Fracture Mechanics Fundamentals and Applications,* 2nd edition, CRC Press 1995

Meyers, M. A. and Chawla, K. K., *Mechanical Behavior of Materials*, 2nd edition, Cambridge University Press 2008

Young, W. C. and Budynas, R. G., *Roarke's Formulas for Stress and Strain*, 7th Edition, McGraw-Hill Publishers 2002

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

General Approach to Report Writing in Metallurgy, Metallurgy Manual Metal 900, Chemistry Unit, latest revision

Chemistry Unit (CU) FBI-Approved Standards for Scientific Testimony and Report Language for Metallurgy, Metallurgy Manual Metal 901, Chemistry Unit, latest revision

Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline, latest revision

Digital Radiography, Metallurgy Manual Metal 303, Chemistry Unit, latest revision

Operation of the SmartScope FOV Video Measurement System, Metallurgy Manual Metal 302, Chemistry Unit, latest revision

Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), Metallurgy Manual Metal 500, Chemistry Unit, latest revision

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Metallurgy Manual Metal 400, Chemistry Unit, latest revision

Metallographic Examinations, Metallurgy Manual Metal 800, Chemistry Unit, latest revision

Operation of Rockwell Hardness Testers, Metallurgy Manual Metal 701, Chemistry Unit, latest revision

Operation of Microhardness Testers, Metallurgy Manual Metal 702, Chemistry Unit, latest revision

Operation of the Instron Model 3382 Universal Testing Machine, Metallurgy Manual Metal 703, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Balances, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Micrometers and Calipers, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Scanning Electron Microscope (SEM)-Energy Dispersive X-ray Spectrometer (EDS), Instrument Operations Systems Support, Chemistry Unit, latest revision

Examinations for Association or Origin, Metallurgy Manual 100, Chemistry Unit, latest revision

Rev. #	Issue Date	History
7	02/18/2020	Revised section 7. Removed reference to x-ray diffraction analysis in section 8.g.
8	07/15/2021	Revised Scope. Minor grammatical changes. Changed "sample" to "specimen", "material", or "object" when appropriate. Added example to Sampling section. Added reference to *Examinations for Association or Origin* for comparisons based on quantitative data. Clarified use of measurement uncertainty and specified the interval for recalculating the repeatability component in Measurement Uncertainty section.

Approval Redact - Signatures on File

Metallurgy Technical Leader: Date: 07/14/2021

Chemistry Unit Chief: Date: 07/14/2021

Appendix A: *Hypergeometric Table*

The hypergeometric table listed below shows the minimum number of samples that need to be analyzed (and yield consistent results) to obtain a 95% confidence level that at least 90% of the population contains a given substance.

Total Number of Units	Number of Units to be Sampled
1-10	All (no inferences)
11-13	10
14	11
15-16	12
17	13
18	14
19-24	15
25-26	16
27	17
28-35	18
36-37	19
38-46	20
47-48	21
49-58	22
59-77	23
78-88	24
89-118	25
119-178	26
179-298	27
299-1600	28
more than 1600	29